## High Resolution Powder Diffraction Studies of Zinc-Loaded Zinc-Exchanged Zeolite A.

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**Introduction**: There has been considerable interest in the formation of ionic clusters encapsulated within zeolites due to the potential they have in areas such as optics and sensors etc. [1,2]. Recent work on the reaction of zinc metal vapour with dehydrated zinc-exchanged zeolite A (Zn-A) has shown that partially reduced zinc-oxide clusters are formed instead of zinc metal clusters [3]. The work presented here is a continuation of that work and looks at a sample with a lower zinc loading level in order to investigate how the zinc-oxide clusters form.

**Methods and Materials**: Zn-A was prepared from Na-A by conventional aqueous ion-exchange methods and was dehydrated under vacuum at 500°C for 12 hours. Zinc metal was then added under an inert atmosphere to give a loading level of one extra zinc atom per unit cell. The reaction took place in a sealed evacuated quartz tube at 350-400°C for 3 days, which resulted in a pale yellow homogeneous powder. For diffraction studies to be carried out a small amount of the sample was packed into a 0.7mm glass capillary under an inert atmosphere and sealed with epoxy resin.

**Results**: The GSAS suite of programs was used for least-squares refinement with the framework positions of McCusker and Seff [4] as the starting model, space group Pm-3m and a lattice parameter of 12Å. After initial refinement difference Fourier maps were used to locate the non-framework atoms. The three most intense peaks in the Fourier map were in similar positions to those of Readman et al. [3] and were therefore assigned as zinc. Inclusion of these sites greatly improved the fit. Difference Fourier maps were again generated and located a site close to that of the non-framework oxygen found previously. Inclusion of this site as oxygen again improved the fit, however, the refined isotropic temperature factor was large and so the non-framework oxygen was displaced slightly to a lower symmetry site. This gave sensible temperature factors and after further fitting of the peak shapes, the refinement converged with $\chi^2$=5.546, $R_{wp}$=3.34%, $R_p$=2.56%, $D_{wd}$=0.540 and $R_{F2}$=4.47.

**Conclusions**: The results here are similar to those of the previous refinement [3], however the fractional occupancies obtained for the zinc sites are different. Further analysis is currently in progress to ascertain the geometry and formula of the species formed in the sodalite cage.

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**References**: G. D. Stucky, J. E. MacDougall, Science, **247**, 699, 1990. [2] P.P. Edwards, P.A. Anderson, J. M. Thomas, Accounts of Chemical Research, **29**, 23, 1996. [3] J.E. Readman, I. Gameson, J.A. Hriljac, P.P. Edwards, P. A. Anderson, Chemical Communications, 595, 2000. [4] L.B. McCusker, K. Seff, Journal of Physical Chemistry, **85**, 405, 1981.

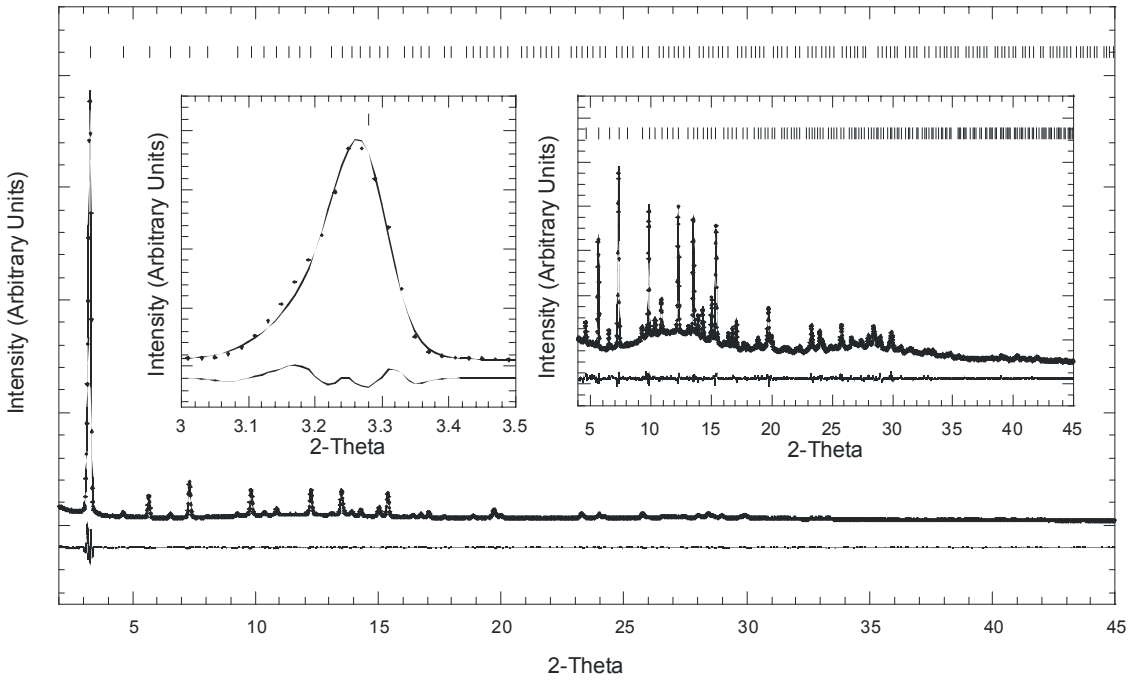


**Figure 1**. Final Rietveld fits showing observed (dots), calculated (straight line) and difference plots.